BRITISH STANDARD

BS 2000-135:
2005
IP 135:2005

Methods of test for petroleum and its products —

Part 135: Determination of rust-preventing characteristics of steam-turbine oil in the presence of water
(Identical with IP 135:2005)

ICS 75.100

BSI
British Standards

National foreword

This British Standard reproduces verbatim IP 135:2005 and implements it as the UK national standard. It supersedes BS 2000-135:1993 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PT/13, Petroleum testing and terminology, which has the responsibility to:

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Energy Institute, 61 New Cavendish Street, London W1G 7AR.
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Summary of pages

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Amd. No.	Date	Comments

Determination of rust-preventing characteristics of steam-turbine oil in the presence of water

This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This standard specifies a method for evaluating the ability of inhibited mineral oils, particularly steam-turbine oils, to aid in preventing the rusting of ferrous parts should water become mixed with the oil. This method can also be used for testing other oils, such as hydraulic oils and circulating oils and provision is also made in the method for testing heavier-than-water fluids.

NOTE 1 - Until 2003, it was customary to run the test for 24 h. A round robin using different test times showed that no statistically significant differences in rating were found, for any sample, between the 4 h and 24 h results. The results of the round robin with modified test duration are held at the Energy Institute.

NOTE 2 - The test duration may, with the agreement of the contracting parties, be for a shorter or longer period.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below.

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

BS 970-1, *General inspection and testing procedures and specific requirements for carbon, carbon manganese, alloy and stainless steels*

2 Principle

300 ml of the oil being tested is placed in a beaker, immersded in an oil bath, fitted with a stirrer and heated to 60°C. A prepared steel specimen is placed in the oil and the oil stirred for 30 min. After 30 min 30 ml of either distilled or synthetic sea water is added and the mixture is maintained at 60°C and stirred for the duration of the test. At the end of the test period the steel specimen is removed and examined without magnifiaction for evidence of rusting. The test procedure is carried out in duplicate.

3 Apparatus

3.1 Oil bath, thermostatically controlled capable of maintaining a temperature in the oil being tested of 60°C ± 1°C, with a cover with holes to accommodate the beakers.

3.2 Beaker glass, tall-form approximately 27 mm in height measured from the inside bottom centre and approximately 70 mm inside diameter measured at the middle with a nominal capacity of 400 ml, see figure 1.

3.3 Beaker cover, glass or methyl methacrylate resin kept in position by suitable means such as a rim or groove with three holes. One for a stirrer, 11,7 mm to 12,2 mm in diameter, with its centre 6,1 mm to 6,6 mm from the centre of the cover. One, on the opposite side of the centre of the cover, for the test specimen assembly, 17,6 mm to 18,1 mm in diameter, with its centre 15,6 to 16,1 mm from the centre of the cover. One 11,7 mm to 12,2 mm in diameter, for a thermometer, with its centre 21,9 mm to 22,5 mm from the centre of the cover, and on a diameter of the cover at right angles to the diameter through the other two holes.

NOTE 3 - An inverted, modified Petri dish makes a suitable cover, as the sides of the dish aid in keeping it in position.

NOTE 4 - Figure 2 shows a methyl methacrylate resin cover for the beaker which has been found to be suitable. An optional feature is shown consisting of a slot, 1,6 mm by 27 mm, which is centred on a diameter of the stirrer hole at right angles to the cover diameter through the specimen hole and stirrer hole. This feature allows

withdrawal of the stirrer while the beaker cover is in place. When testing synthetic fluids, it is recommended that the beaker cover and specimen holder should be made from chemically resistant material such as polymonochlorotrifluoroethane (PCTFE).

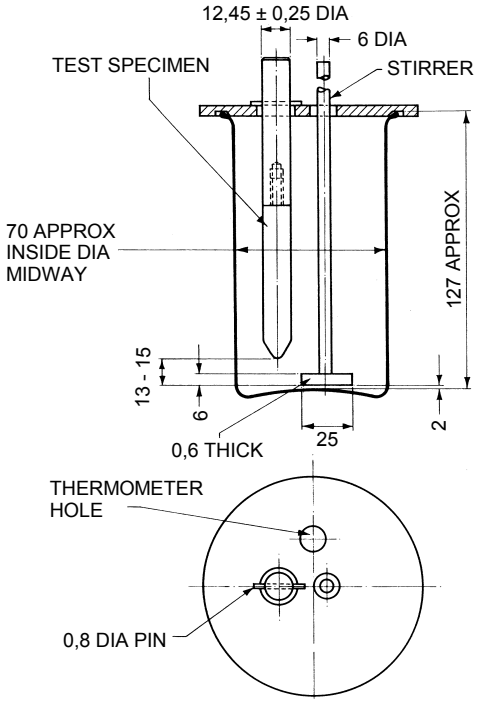

Figure 1. Apparatus for rusting test

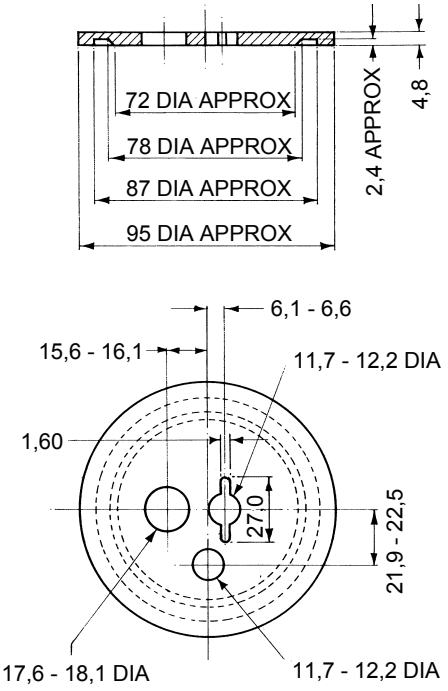

Figure 2. Beaker cover

3.4 Stirrer, either of stainless steel or heat-resistant glass (e.g. borosilicate glass) in the form of a flat bladed inverted 'T' approximately 25 mm by 6 mm by 0,6 mm attached to a 6 mm rod in such a way that the blade is symmetrical with the rod and has its flat surface in the vertical plane.

3.5 Modified stirrer, to aid the mixing of heavier-than-water test fluids the stainless steel stirrer described in 3.4 may be fitted with an auxiliary blade attached to the shaft. The auxiliary blade shall be of stainless steel approximately 19 mm by 12,7 mm by 0,6 mm positioned on the stirrer shaft, so that the bottom edge of the auxiliary blade is approximately 60 mm above the top edge of the fixed lower blade, and so that the flat surfaces of both blades are in the same vertical plane, see figure 3.

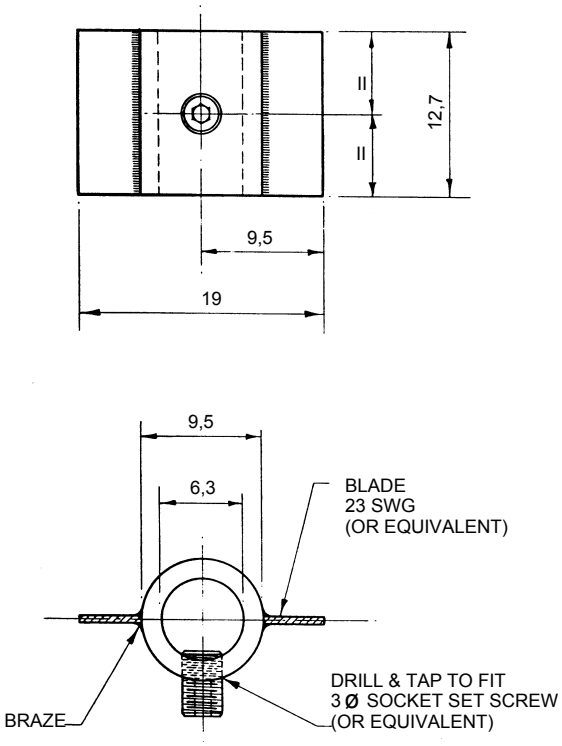

Figure 3.

3.6 Stirring apparatus, any convenient form of stirring apparatus capable of maintaining a speed of 1 000 rpm ± 50 rpm.

3.7 Grinding and polishing equipment, metal working aluminium oxide abrasive cloth, closed coat on a denim backing, European grades P150J (97µm) and P280J (53µm) conforming to BS 871: 1981 or equivalent.

NOTE 5 - The approximate corresponding American Grades are A150 and A240 respectively.

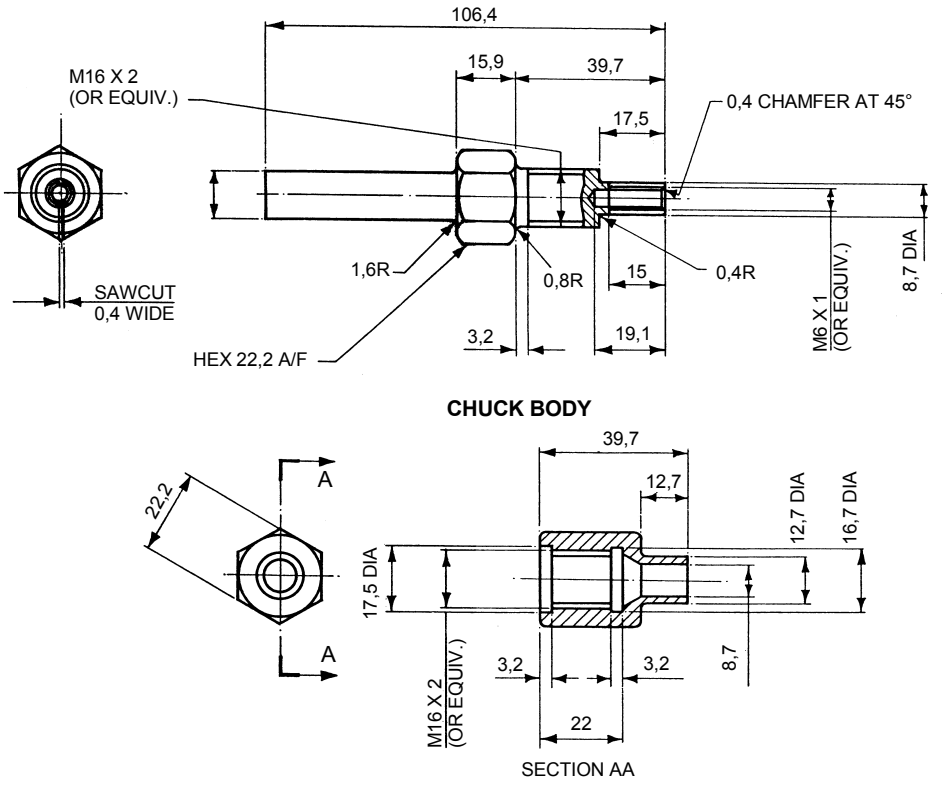

Figure 4. Chuck for polishing test specimens

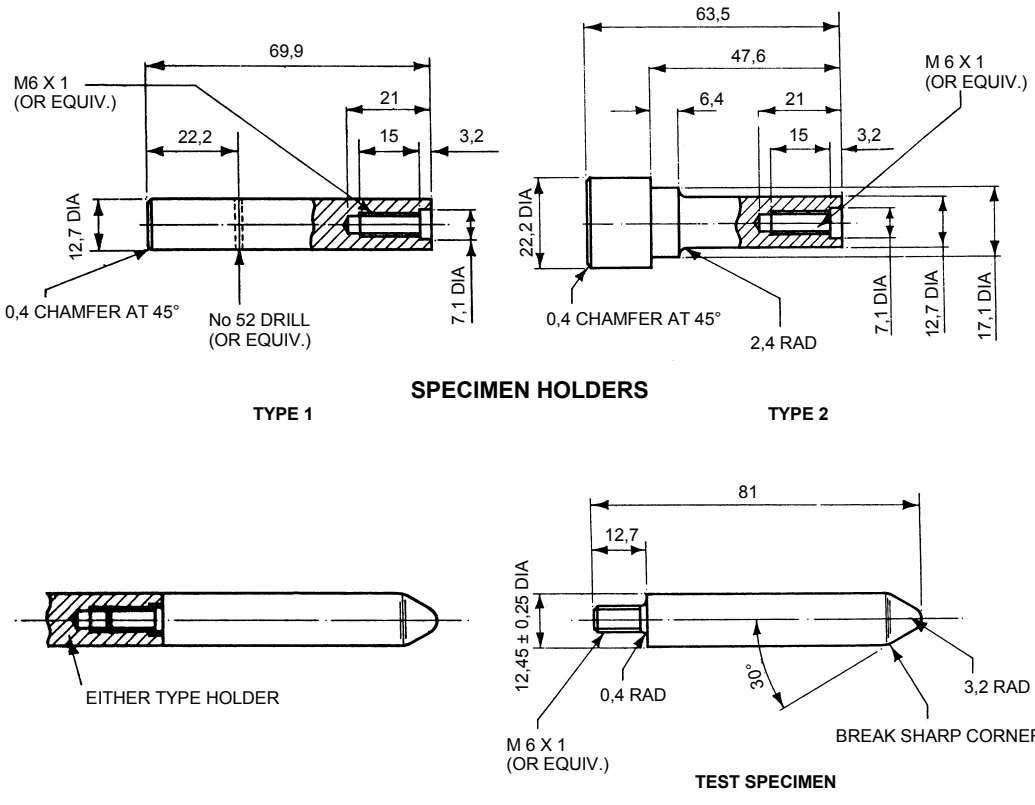

Figure 5.

Table 1

Salt	purity % m/m	concentration g/l
Sodium chloride NaCl	99,9 (after ignition)	25,54
Magnesium chloride 6-hydrate $MgCl_2.6H_2O$	98,0	11,10
Sodium sulphate Na_2SO_4	99,5	4,09
Calcium chloride 2-hydrate $CaCl_2.2H_2O$	99,5	1,54
Potassium chloride KCl	99,5	0,69
Sodium hydrogen carbonate $NaHCO_3$	99,5	0,20
Potassium bromide KBr	99.5 (after drying)	0,10
Boric acid H_3BO_3	99,8	0,03
Strontium chloride 6-hydrate $SrCl_2.6H_2O$	99,0	0,04
Sodium fluoride NaF	99,0	0,003

3.8 Drill or lath, fitted with a suitable chuck see figure 4, for holding the test specimen and capable of rotating the specimen at a speed of 1 750 rpm ± 50 rpm.

3.9 Test specimen, carbon steel conforming to 070 M20 of BS 970-1, see notes 6 and 7. Dimensions when new, diameter 12,45 mm ± 0,25 mm, length 68,0 mm ± 0,25 mm, exclusive of the threaded portion which screws into the plastics holder, and tapered at one end as shown in figure 5.

NOTE 6 - Grade 10180 of ASTM Specification A108, for Cold-finished Carbon Steel Bars and Shafting Steel, and European Steel C22E number 1.1151 are equivalent grades.

NOTE 7 - If the specified steel is not available, an equivalent steel which is found to be satisfactory by comparative testing using IP 135 may be used.

3.9 Test specimen holder, either methyl methacrylate resin or for testing synthetic fluids chemically resistant material such as polymonochlorotrifluoroethane (PCTFE). With dimensions as shown in figure 5 (two types of holders are illustrated).

3.10 Balance, capable of weighing to 0,1 mg.

3.11 Volumetric flask, capacity 1l.

3.12 pH Meter

3.13 Thermometer, capable of reading to 60°C ± 1°C.

4 Reagents and materials

Use only chemicals and reagents of recognised analytical grade and water conforming to grade 3 of ISO 3696.

4.1 2,2,4-Trimethylpentane, conforming to IP Standard Reference Liquids Specification as given in Appendix B.

4.2 Detergent cleaning solution.

4.3 Sodium carbonate Na_2CO_3 solution 0,2 M.

4.4 Synthetic sea water.

4.4.1 Comprising of salts with purities and concentrations as listed below table 1.

4.4.2 Synthetic sea water preparation.

NOTE 8 - This method avoids any precipitation in concentrated solutions with subsequent uncertainty of complete re-solution.

Using the salts and distilled water, prepare the following stock solutions:

Stock solution 1
Weigh the following amounts of the salts:

$MgCl_2.6H_2O$	555 g
$CaCl_2.2H_2O$	77 g
$SrCl_2.6H_2O$	2 g

Dissolve in distilled water and place into a 1l volumetric flask (3.11) and make up to the mark.

Stock solution 2
Weigh the following amounts of the salts:

KCL	69 g
$NaHCO_3$	20 g
KBr	10 g
H_3BO_3	3 g
NaF	0,3 g

Dissolve in distilled water and place into a 1l volumetric flask (3.11) and make up to the mark.

Dissolve 24,54 g of NaCl and 4,094 g of Na_2SO_4 in a few hundred millilitres of distilled water, add 20 ml of stock solution 1 and 10 ml of stock solution 2 and dilute to 1l. Stir the 1l batch and insert the electrodes of the pH meter (3.12) add the sodium carbonate solution (4.3) until the pH is between 7.8 and 8.2. The resulting solution should be clear and bright, and free from residual salt.

NOTE 9 - Between 0,1 ml to 0,2 ml of the Na_2CO_3 solution will normally be required.

Store the solutions in a cool, dark environment. Inspect prior to use and ensure that the solutions remain free from salt deposits or algal growth.

5 Test specimen preparation

5.1 For each test oil, prepare two steel specimens in accordance with 5.2 and 5.3.

NOTE 10 - These may either be new test specimens or ones used in a previous test.

When making a check test, the steel specimen that showed rust shall not be reused. Specimens that repeatedly show rust in tests of various oils may be imperfect. Such specimens shall only be used with oils known to pass the test. If rusting occurs in repeat tests, these specimens shall be discarded.

5.2 The specimen shall be subjected to preliminary grinding, unless it has been used previously and is free from rust or other irregularities, in which case it shall be subjected to final polishing in accordance with 5.3. Previously used rust free specimens shall be stored in 2,2,4 trimethylpentane.

If the specimen is new, or if any part of its surface shows rust or other irregularities, clean it with 2,2,4-trimethylpentane and grind with P150J aluminium oxide cloth to remove all irregularities, pits and scratches, as determined by visual inspection. Perform the grinding by mounting the specimen in the chuck of the grinding and polishing apparatus and turning it at a speed of 1 750 rpm ± 50 rpm while the P150J aluminium oxide cloth is applied. Although old P150J aluminium oxide cloth may be used to remove rust or irregularities, the grinding shall be completed with new cloth. Proceed at once with the final polishing with P280J aluminium oxide cloth, or remove the specimen from the chuck and store in 2,2,4-trimethylpentane until needed.

The specimen shall not be touched with the hands at any stage after cleaning with the 2,2,4-trimethylpentane (which precedes either preliminary grinding or final polishing) until the test is completed. Plastic forceps, clean gloves or a clean, lintless cloth shall be used.

Discard reused specimens when the diameter is reduced to approximately 9,5 mm.

5.3 Just before the test is to be made, subject the specimen to final polishing with P280J aluminium oxide cloth. If the preliminary grinding has just been completed, stop the motor which rotates the specimen. Otherwise, remove the specimen from the 2,2,4-trimethylpentane in which it is stored, dry with a clean cloth and place in the chuck.

Rub a new piece of P280J aluminium oxide cloth longitudinally over the static specimen until the rounded end, and the entire surface, show visible scratches. Rotate the specimen at a speed of 1750 rpm ± 50 rpm and polish with a strip of P280J aluminium oxide cloth by wrapping it halfway around the specimen and applying a firm but gentle downward pull to the loose ends of the cloth for

about 1 min to 2 min moving the cloth along the pin surface, so as to produce a uniform, finely scratched surface free from longitudinal scratches. Carry out the final stage of the polishing with new cloth, ensuring that fresh cloth surface is exposed to the pin throughout. Approximately 10-12 passes with the cloth should be adequate, subject to visual inspection under good light.

To ensure that the flat shoulder (that portion of the specimen perpendicular to the threaded stem) is free of rust, this area should be polished. This may be achieved by holding a strip of P280J aluminium oxide cloth between the chuck and the shoulder while rotating the specimen for a brief period.

Remove the specimen from the chuck without touching with the fingers, either wipe lightly with a clean, dry, lintless cloth or tissue or brush the specimen lightly with a camel-hair brush, attach to the plastics holder, and immediately immerse in the oil to be tested, see note 12.

NOTE 12 - This may be either the hot oil sample see 6.1.1 or a clean test tube containing a portion of cold oil sample. The specimen may be removed later from this tube and allowed to drain briefly before being placed in the hot oil.

6 Procedure

Tests shall be conducted in duplicate.

6.1 Procedure using distilled water

6.1.1 Clean the beaker with detergent cleaning solution, wash thoroughly with distilled water and dry in an oven. If a glass beaker cover and/or stirrer are to be used, clean by the same procedure. To clean a stainless steel stirrer and a methyl methacrylate resin cover, use 2,2,4-trimethyl-pentane, wash thoroughly with hot water and finally with distilled water, and dry in an oven at a temperature not exceeding 65°C for the cover. Pour 300 ml of the oil to be tested into the beaker and place the beaker in the oil bath held at a temperature that will maintain 60°C ± 1°C in the oil sample. The beaker shall be inserted into a hole in the bath cover and suspended in the hole with the beaker rim resting on the bath cover. The oil level in the bath shall not be below the oil level in the test beaker. Cover the beaker with the beaker cover with the stirrer in position in the correct opening. Adjust the stirrer so that the shaft is 6 mm off centre in the beaker containing the oil sample, and the blade is within 2 mm of the bottom of the beaker. Then suspend a thermometer through the hole in the cover intended for that purpose, so that it is immersed to a depth of 57 mm, see note 13. Start the stirrer and when the thermometer reading reaches 60°C ± 1°C, insert the steel specimen prepared in accordance with clause 5.

NOTE 13 - When analysing multiple samples of a similar nature that are introduced into a thermostatically controlled bath at approximately the same time (that is, individual samples being analysed as a batch)data collected have shown that it is not necessary to suspend a temperature measuring device through the hole in the cover intended for that purpose in each of the samples, since a thermostatically controlled bath is capable of maintaining the proper bath temperature within the allowed limits at each of the sample beaker locations. As such, it is permissible to suspend a temperature measuring device through the hole in the cover intended for that purpose in as few as one of the sample being analysed, immersed to a depth of about 56 mm. The temperature reading measured in the sample beaker location selected is the basis for determining when the temperature reached 60 ± 1°C in order to begin stirring each of the beakers and inserting the steel test rods. Determining the temperature profile of the bath is strongly recommended in this circumstance to ensure adequate temperature control.

6.1.2 Insert the test specimen through the specimen hole in the beaker cover and suspend so that its lower end is 13 mm to 15 mm from the bottom of the beaker. Either type of plastics specimen holder, see figure 5 may be used. The hole through which the specimen is suspended shall be unobstructed, see figure 1.

6.1.3 Continue stirring for 30 min to ensure complete wetting of the steel specimen. With the stirrer in motion, remove the thermometer temporarily, and add 30 ml of distilled water through the hole for the thermometer, discharging the water onto the bottom of the beaker. Replace the thermometer. Continue stirring at a speed of 1 000 rpm ± 50 rpm for the required time from the time the water was added, maintaining the temperature of the oil water mixture at 60°C ± 1°C, see note 1. At the end of the required period stop the stirrer and remove the specimen. Allow the specimen to drain briefly, and then wash with 2,2,4-trimethylpentane until free from oil residue. Allow the 2,2,4-trimethylpentane to run down the specimen from the holder to ensure that loose rust is not removed.

NOTE 14 - If desired, the specimen may be preserved by lacquering.

NOTE 15 - If the test duration is 24 h, then in general observations of rusting at the end of 12 h testing time are indicative as to whether the oil will pass or fail.

6.2 Procedure using synthetic sea water

Repeat the procedures of 6.1.1 to 6.1.3, using the synthetic sea water (4.4) in place of distilled water.

6.3 Procedure for testing heavier-than-water fluids

For heavier-than-water test fluids, the stirring action provided by the stirrer (3.4) is not sufficient to thoroughly mix the water and test sample. Also the beaker cover and test specimen holder may be subject to chemical attack.

Proceed in accordance with clauses 6.1 or 6.2 with the following exceptions.

a) To resist chemical attack, use a polychlorotrifluoroethylene beaker cover and polytetrafluoroethane specimen holder.

b) Use the modified stirrer (3.5).

7 Examination of the test specimen

Specimens shall be inspected without magnification under normal light, i.e. an illumination of approximately 650 lx, see note 16.

NOTE 16 - 650 lx equates approximately to strong daylight, or within 1 m of a 40 W fluorescent tube.

A rusted specimen shall be one on which, any rust spot or streak on the body of the specimen is visible by this procedure. Do not rate the tapered point, or the shoulder area immediately adjacent to the holder.

For the purpose of this test method, rust is an area of corrosion of the test surface that is identified by colour, and is confirmed by the presence of pits or roughness if the surface is gently wiped with a lintless or tissue paper cloth. Neither surface discolouration, nor specks that can be easily removed with a lintless or tissue paper cloth with no evidence of pitting or roughness shall be considered to be rust. Some formulations are known to leave grey/white deposits due to additive or salt deposition; these too are not considered to be rust unless indicated by colour.

8 Expression of results

An oil shall be reported as passing the test if both specimens are rust free at the end of the test period. An oil shall be reported as failing the test if both specimens are rusted at the end of the test period.

NOTE 17 - An indication of the degree of rusting occurring in this test may be desired. For uniformity in such cases, use of the following classifications of rusting severity is recommended:

Light rusting - Rusting confined to not more than six spots, each of which is 1 mm or less in diameter.

Moderate rusting - Rusting in excess of light rusting, but confined to less than 5 % of the surface of the specimen.

Severe rusting - Rusting covering 5 % or more of the surface of the specimen.

If one specimen is rusted while the other is free of rust, tests on two additional specimens shall be made see 5.1.

If either of these latter specimens show rusting, the oil shall be reported as not passing the test. If neither of these latter specimens shows rusting, the oil shall be reported as passing the test.

9 Precision

Since the results of this test are intended only to give a pass/fail rating to the oil being tested, no statement will be made about either the precision or the accuracy.

10 Report

The test report shall include:

a) the type and identification of the product being tested;

b) a reference to the test method and procedure used; e.g. IP 135 stating whether distilled or synthetic water was used;

c) the duration of the test;

d) the full name and address of the test laboratory;

e) the date of the test.

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Energy Institute

61. New Cavendish Street
London
W1G 7AR

Tel: +44 (0)20 7467 7100
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